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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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Indian Standard

METHOD FOR DETERMINATION OF IRON AND CHROMIUM IN TEXTILES

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Indian Standard

METHOD FOR DETERMINATION OF IRON AND CHROMIUM IN TEXTILES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 17 June 1968, after the draft finalized by the Chemical Methods of Tests Sectional Committee had been approved by the Textile Division Council.

0.2 Iron and chromium are present in large quantities in textiles, dyed in mineral khaki. Mineral khaki dyed material is used mainly by Defence and civilians as well for making uniforms. The iron and chromium content of the fabric would give an indication regarding the amount of mineral khaki present in the textile material. This standard would be useful for the purpose of determining iron and chromium present in the textile material.

0.3 Considerable assistance has been derived from Joint Services Specification JSS-1250 'Methods of tests for textile' issued by the Ministry of Defence.

0.4 This edition 1.1 incorporates Amendment No. 1 (June 2002). Side bar indicates modification of the text as the result of incorporation of the amendment.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes method for determination of iron and chromium present in textile materials, especially dyed in mineral khaki and olive green shades.

2. PRINCIPLE

2.1 A known amount of test sample is ashed and the amount of iron and chromium is determined as their oxides (Fe_2O_3 and Cr_2O_3) and expressed as the percentage of the conditioned weight of the test sample.

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*Rules for rounding off numerical values (*revised*).

3. SAMPLING

3.1 Sampling for Yarn

3.1.1 *Lot (Yarn)* — The quantity of yarn from the same source shall constitute the lot. If the lot contains more than 200 kg of yarn, it shall be divided into sub-lots, each weighing 200 kg or less.

3.1.2 *Test Sample* — Unless otherwise agreed to between the buyer and the seller, 15 increments each approximately weighing 10 g shall be taken from different parts of each sub-lot so that a representative test sample is obtained. All the increments thus collected shall be thoroughly mixed.

3.2 Sampling for Fabrics

3.2.1 *Lot (Fabric)* — The quantity of fabrics manufactured essentially under uniform conditions shall constitute a lot.

3.2.2 *Gross Sample* — Unless otherwise agreed to between the buyer and the seller, the number of pieces to be selected from a lot to constitute gross sample shall be as given below:

Lot Size	*Sample Size*
Up to 100	3
101 ,, 300	4
301 ,, 500	5
501 and above	7

3.2.3 *Test Sample* — From each piece in the gross sample about 25 g of fabric shall be taken out from at least two different parts to constitute test sample. The parts shall then be cut into further smaller pieces and thoroughly mixed.

4. PREPARATION OF TEST SPECIMEN

4.1 Cut the test sample into small pieces. Mix all the pieces thoroughly. Draw at least 3 test specimens from among these pieces such that each specimen weighs 5 g.

5. CONDITIONING OF TEST SPECIMEN

5.1 Prior to test, the test specimen shall be conditioned for 24 hours to moisture equilibrium in a standard atmosphere at 65 ± 2 percent RH and 27° ± 2°C temp (*see* IS : 196-1966*).

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*Atmospheric conditions for testing (*revised*).

6. APPARATUS

6.1 Porcelain Crucible

6.2 Beakers

7. REAGENTS

7.1 Quality of Reagents — Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS : 1070-1960*) shall be used where the use of water or distilled water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

7.2 Hydrogen Peroxide — 6 percent (*w*/*w*) (or 20 volumes).

7.3 Sodium Hydroxide Solution — 20 percent (*w*/*v*).

7.4 Dilute Hydrochloric Acid — equal volumes of concentrated hydrochloric acid and water.

7.5 Dilute Sulphuric Acid — 10 percent (*w*/*v*).

7.6 Mixed Acid Solution — prepared by mixing 150 ml of concentrated sulphuric acid and 150 ml of phosphoric acid and made up to 1 litre with water.

7.7 Ferrous Ammonium Sulphate Solution — N/10.

7.8 Potassium Dichromate Solution — (a) N/10, and (b) N/20.

7.9 Stannous Chloride Solution — 5 percent (*w*/*v*).

7.10 Mercuric Chloride Solution — saturated.

7.11 Indicator Solution — prepared by dissolving 0.2 g of barium diphenylamine sulphonate in 100 ml of water.

7.12 Potassium Permanganate Solution — (a) saturated, and (b) N/20.

7.13 Potassium Bisulphate — anhydrous.

7.14 Potassium Iodide — crystals.

7.15 Starch Indicator — 1 percent solution.

7.16 Sodium Thiosulphate Solution — N/20.

7.17 Reinhardt Solution — prepared by dissolving 200 g of manganese sulphate in 1 000 ml of water to which is added a cooled mixture of 400 ml of concentrated sulphuric acid, 1 200 ml of water and 400 ml of syrupy phosphoric acid.

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*Specification for water, distilled quality (*revised*). (Since revised).

7.18 Nitric Acid — concentrated.

7.19 Sodium/Potassium Chlorate — crystals.

7.20 Ammonium Hydroxide

7.21 Ammonium Chloride — crystals.

8. PROCEDURE

8.1 Condition the test specimens to moisture equilibrium in standard atmosphere (*see* **5**) and weigh each test specimen accurately.

8.2 Put a test specimen in porcelain crucible (*see* Note), ash it in a muffle furnace at 800°C for 15 minutes and fuse the ash over a bunsen flame to clear melt with 10 g of potassium bisulphate. Dissolve the melt in 100 ml of hot water and add 100 ml of hydrogen peroxide. Add enough amount of sodium hydroxide solution till the solution is slightly alkaline. Boil the solution gently for 5 minutes and filter it through Whatman No. 54 or its equivalent filter paper. Wash the precipitate with hot water. Collect the filtrate and the washings.

NOTE — If the amount of iron and chromium compounds which have been fixed properly on the fabric is to be determined the water-soluble compounds of iron and chromium (which have not been properly fixed) should be removed by the following procedure:

'Before ashing, put the test specimen in a 250-ml beaker. Add 100 ml of distilled water and boil it for 15 minutes. Wash the test specimen thoroughly, first with warm water and then with cold water. Dry the test specimen and follow the procedure given in **8.2**.

8.3 Dissolve the precipitate obtained as in **8.2** in 10 ml of hot dilute hydrochloric acid. Wash the filter paper thoroughly with distilled water till the washings are free from iron. Add 5 ml of hydrogen peroxide solution and reprecipitate iron with sodium hydroxide solution as in **8.2**. Boil it for 5 minutes. Filter off the precipitate and wash it till free from chromium.

8.4 Combine the filtrate as obtained in **8.2** and **8.3**.

8.5 Determination of Chromium

8.5.1 *First Method*

8.5.1.1 Concentrate the filtrate obtained as in **8.4** and neutralize it with dilute sulphuric acid. Add 10 ml of dilute sulphuric acid in excess. Add 25 ml of N/10 ferrous ammonium sulphate solution followed by 20 ml of mixed acid solution and 1 ml of indicator solution. Titrate the mixture against N/10 potassium dichromate solution to the first permanent blue colour.

8.5.1.2 Carry out a blank test by following the procedure given in **8.5.1.1** using distilled water instead of the filtrate obtained as in **8.4**.

8.5.1.3 Calculate the percentage of chromium as chromium oxide (Cr_2O_3) by the following formula:

$$A = \frac{(V_2 - V_1) \times 0.253\,4}{W}$$

where

A = percent by weight of chromium as Cr_2O_3,

V_2 = volume in ml of N/10 potassium dichromate required for blank (*see* **8.5.1.2**),

V_1 = volume in ml of potassium dichromate required for the test (*see* **8.5.1.1**), and

W = weight in g of the conditioned test specimen (*see* **8.1**).

8.5.2 *Second Method*

8.5.2.1 Take the filtrate obtained as in **8.4** and make up the volume to 250 ml. Take 50 ml of the diluted solution in a flask and neutralize it with dilute sulphuric acid and add 10 ml of dilute sulphuric acid in excess. Cool the solution to room temperature. Add 2 g of potassium iodide crystals and keep the flask stoppered for 5 minutes in dark place. Titrate the liberated iodine against standard sodium thiosulphate solution using starch as an indicator.

8.5.2.2 Calculate the percentage of chromium as its oxide (Cr_2O_3) by the following formula:

$$P = \frac{A \times B \times 25.34}{2\ W}$$

where

P = percent by weight of chromium as Cr_2O_3,

A = volume in ml of sodium thiosulphate solution required for the test,

B = strength of the sodium thiosulphate used, and

W = conditioned weight in g of the specimen (*see* **8.1**).

8.5.3 *Third Method*

Put a test specimen in porcelain crucible (*see* Note), and ash it in a muffle furnace at 800°C for 15 minutes. Dissolve the ash in the beaker in minimum amount of concentrated nitric acid and then add sodium/potassium chlorate (5 g) with small quantity of water. Evaporate the solution to just dryness on a sand bath and then add to it 100 ml of hot water to bring the residue into solution. To the above solution add solutions of ammonium chloride or ammonium sulphate and ammonium hydroxide to precipitate the iron. The precipitate is filtered and washed repeatedly with hot water till it is free from chromium.

NOTE — If the amount of iron and chromium compounds which have been fixed properly on the fabric is to determine the water soluble compounds of iron and chromium (which have not been properly fixed) should be removed by the following procedure:

Before ashing, put the test specimen in a 250-ml beaker. Add 100 ml of distilled water and boil it for 15 minutes. Wash the test specimen thoroughly, first with warm water and then with cold water. Dry the test specimen and follow the procedure given in **8.2**.

8.6 Determination of Iron

8.6.1 Dissolve the precipitate obtained as in **8.3** in hot dilute hydrochloric acid. Boil the solution and add stannous chloride solution drop-wise while boiling until the solution becomes colourless. Cool the solution to room temperature. Add 5 ml of mercuric chloride solution (*see* Note).

NOTE — Excess of stannous chloride should not be added as it would require more of mercuric chloride resulting in the precipitation of mercurous chloride which would interfere in the titration.

8.6.2 *First Method*

8.6.2.1 Add 20 ml of mixed acid solution to the solution obtained as in **8.6.1**. Add 1 ml of indicator and titrate against N/20 potassium dichromate solution to the first permanent blue colour.

8.6.2.2 Calculate the percentage of iron as its oxide (Fe_2O_3) by the following formula:

$$B = \frac{V_3 \times 0.3\,992}{W}$$

where

B = percent by weight of iron as Fe_2O_3,
V_3 = volume in ml of N/20 potassium dichromate solution required for the test (**8.6.2.1**), and
W = weight in g of the conditioned test specimen (*see* **8.1**).

8.6.3 *Second Method*

8.6.3.1 Transfer the solution obtained as in **8.6.1** (after 5 to 10 minutes) to a large porcelain dish. Add to this 25 ml of Reinhardt solution and 400 ml of water. Rinse with a moderate amount of water and titrate against N/20 potassium permanganate solution with a constant stirring to a definite pink tint.

8.6.3.2 Carry out a blank titration on the reagents.

8.6.3.3 Calculate the percentage of iron as its oxide (Fe_2O_3) by the following formula:

$$B = \frac{(V_5 - V_4) \times 0.3\,992}{W}$$

where

B = percent by weight of iron as Fe_2O_3,

V_5 = volume in ml of N/20 potassium permanganate (required for the test),

V_4 = volume in ml of N/20 potassium permanganate (required for blank), and

W = weight in g of the conditioned test specimen.

8.7 Repeat the procedure with the remaining test specimens.

9. REPORT

9.1 Report individually the values obtained as in **8.5.1.3** or **8.5.2.2** and **8.7**, as the percentage of chromium as its oxide (Cr_2O_3) and the values obtained as in **8.6.2.2** or **8.6.3.3** and **8.7** as percentage of iron as its oxide (Fe_2O_3) in the test specimens.

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